

**REMARKS**

Applicants acknowledge the Examiner's careful, independent review of their application and offer the foregoing amendments and the following remarks in an effort to place the application in condition for allowance.

**An amendment to the substitute specification:**

Applicants amended their substitute specification to correct the spelling of Tosoh. It is respectfully requested that this clerical amendment be entered.

**The Amendment to Claim 5:**

Applicants amended claim 5 based on the substitute specification at page 14, lines 4-16. The original and substitute specification each disclose MFRR. This amendment avoids new matter. It is offered to advance prosecution and resolve issues. Entry is therefore respectfully solicited.

**Attachments:**

Attached to this Amendment are copies: a brochure of Tosoh Finhechem, JIS K 7210, ASTM-D1238, U.S. Patent No. 6,355,748B1 and U.S. Patent No. 6,787,616B2. The materials provide information to confirm the catalyst component in Applicants' Comparative Example 5 and to assist the Examiner in re-assessing the applied art in view of Applicants' remarks.

**Clarification Regarding Comparative Example 5:**

Applicants acknowledge the statement in the Office Action that “Applicants’ Comparative Example 5 discloses PMOA solution was used.” This may result from an inadvertent transposition of two letters in the remarks presented in the July 7, 2005 Amendment as explained hereinbelow. Applicants regret any inconvenience to the Examiner.

The statement should *correctly* read “Applicants’ Comparative Example 5 discloses **PMAO** solution was used. Applicants respectfully call attention to their prior Amendment at page 6, middle of the page where the statement inadvertently was typed as “Applicants’ Comparative Example 5 discloses PMOA solution was used (substitute specification page 36, lines 6-7; original specification page 36, lines 14-15).” Emphasis added. Applicants again respectfully point out the statement should correctly have read “Applicants’ Comparative Example 5 discloses PMAO solution was used...” *not* PMOA. The cited passages from the original and substitute specification each refer to PMAO, not PMOA.

This is independently corroborated by the attached brochure from Tosoh Finechem, as well as by U.S. Patent 6,355,748B1 at column 11, lines 25-40 and by U.S. Patent No. 6,787,616B2 at column 19, lines 42-43 and column 10, lines 2-3, as examples. From these exemplary documents it will be appreciated that PMAO was used in Comparative Example 5, and that it’s an aluminoxane (*e.g.*, methyl aluminoxane).

**Traversing the Rejections:**

Applicants respectfully traverse the rejection of claims 5-8 under 35 U.S.C. §102(b) as anticipated by, or in the alternative, obvious under 35 U.S.C. §103(a) over U.S. Patent No. 5,849,653 (Dall'Occo et al.).

Applicants also traverse the rejection of claims 1-4 under 35 U.S.C. §102(b) as anticipated by, or in the alternative, obvious under 35 U.S.C. §103(a) over U.S. Patent No. 5,374,700 (Tsutsui et al.).

Applicants respectfully request reconsideration and withdrawal of the rejections and also of their respective rationales as urged in the Office Action.

It appears that the Examiner is still of the opinion that the catalyst in the cited references is substantially identical to one used in the present invention to prepare the ethylene and  $\alpha$ -olefin copolymer, and based on that hypothesis, has concluded the references disclose a resulting ethylene and  $\alpha$ -olefin co-polymer possessing the claimed properties.

**The catalysts are, in fact, different.**

Applicants respectfully submit there is no *prima facie* case of obviousness, nor is there basis for the anticipation rejection.

The Tsutsui et al. reference describes a component (B) which is known to those skilled in the art as an aluminoxane as seen from column 8, lines 51-56. As disclosed in the Tsutsui et al. reference, aluminoxane can be produced, for instance, by allowing an organoaluminum to react with water or a salt that contains water. Applicants invite the Examiner's attention to column 8, lines 59-65.

The Dall'Occo et al. reference also describes an aluminoxane compound. Even if, *arguendo*, in the Dall'Occo et al. reference, the component (B) comprised of an organometallic aluminum compound and water is used, it would have been apparent to those skilled in the art that the resulting compound is converted *in situ* to an aluminoxane. This is also independently seen from the above-discussed Tsutsui et al. reference.

On the other hand, the present claimed copolymer of ethylene and  $\alpha$ -olefin can be prepared using a different catalyst as disclosed in the present specification at page 17, lines 18-19, whereby both the copolymer is described and is enabled. As the present specification discloses, in a method of feeding the components of a metallocene olefin polymerization catalyst that may be used to produce an ethylene- $\alpha$ -olefin copolymer according to the present invention, the components can be fed, for example, using (under) an inert gas such as nitrogen, argon and the like, or hydrogen, ethylene and the like under condition of no water, specification at page 17, third full paragraph, and the original specification at page 17, third full paragraph (last one before end of page), as examples. At page 16, Applicants' specification discloses an organoaluminum compound is preferably is triisobutylaluminum or tri-n-octylaluminum as disclosed in the substitute specification, page 16, penultimate

paragraph, and original specification, page 16, lines 2 and 3 from the bottom. Accordingly, it will be appreciated by those skilled in the art that a catalyst useful in making the copolymers herein is not necessarily the same as either of the references.

Applicants respectfully suggest that the premise to each rejection should be reconsidered and the rejections withdrawn.

**Contrary to the Office Action, Applicants have provided evidence to rebut the premise to the rejection:**

The Office Action includes the statement that “[h]owever, applicants fail to provide any experimental data to support that the polymers obtained according to the prior art (the use of aluminoxane) will result in a polymer having MFR, MT,  $[\eta]$  and A not meeting the claimed equations.” *See*, Office Action, page 5.

Applicants respectfully request reconsideration and withdrawal of the rejection. Even if there is a *prima facie* case of either anticipation or obviousness, which is not conceded, Applicants respectfully submit the statement in the Office Action is mistaken.

Applicants respectfully submit Comparative Example 5 **rebutts** the conclusion that the catalyst according to the references could and necessarily would catalyze a copolymerization to make the copolymers as claimed herein.

Applicants respectfully submit their Comparative Example 5 reports the ethylene copolymer as claimed could not be obtained using aluminoxane as one component of the

metallocene catalyst corresponding to the cited references. Applicants respectfully invite the Examiner's attention to their Comparative Example 5, substitute specification commencing at page 35 at the third line from the bottom, and the original specification page 36, starting at line 6. According to Comparative Example 5, a copolymer did not satisfy the claimed properties. The copolymer gives a film having a lot of fish eyes. The copolymer gives a film having high haze. The film has insufficient appearance. Applicants' Comparative Example 5 discloses *PMAO* solution was used. The Examiner can confirm this by inspecting the substitute specification at page 36, such as at lines 6-7 and the original specification page 36, such as at lines 14-15. The original and substitute specification each disclose that according to Comparative Example 5, the relationship in Formula (3) and the relationship in Formula (4), as an example, are not satisfied. *See, e.g.*, specification at page 42. The film that is made according to Comparative Example 5 has fish eyes. *Id.*

It is also clear from Table 1 in the specification that the ethylene copolymer obtained does not satisfy at least the relationship (3) when aluminoxane is used (Comparative Example 5), instead of an organoaluminum such as triisobutylaluminum as in Examples 1-5.

Table 1 additionally makes it clear that the ethylene copolymers of Comparative Examples 1-5 that do not satisfy the relationship (3) are inferior in transparency. Stated another way, films of such copolymers have a high haze value (%).

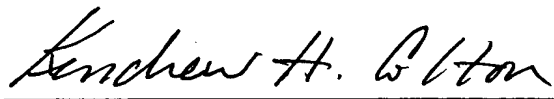
Furthermore, the MFRR of 60 or more in amended claim 5 is neither disclosed in nor would it have been suggested by Dall'Occo. The copolymers as disclosed in Examples 1 to 11 of Dall'Occo have an F/E within the range of 21.7 to 38.3 as the Examiner will see from

Dall'Occo's Table 1 (spanning the top of columns 17-18). The F/E is similar to MFRR as seen from column 8, lines 19-26. Herein, F/E may be said to correspond to MFRR because it is well known that the test method according to ASTM D-1238 (copy attached) almost corresponds to that of JIS K-7210 (copy attached.). Dall'Occo refers to ASTM D-1238 at column 8, lines 23 and 25. Furthermore, Dall'Occo does not provide any motivation to produce ethylene copolymers having an F/E ratio suggestive of the claim 5 copolymers having an MFRR of 60 or more.

**Conclusion:**

Applicants respectfully submit their application is in condition for allowance and respectfully solicit a Notice of Allowance.

Respectfully submitted,



Kendrew H. Colton  
Registration No. 30,368

Date: December 20, 2005

*OFFICIAL CORRESPONDENCE TO*  
*Customer No. 42798*  
FITCH, EVEN, TABIN & FLANNERY  
1801 K Street, NW, Suite 401L  
Washington, DC 20006-1201  
Tel: (202) 419-7000  
Fax: (202) 419-7007



**TOSOH FINECHEM CORPORATION**

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**E**

Alkyl Aluminum Polymerization Catalysts Organic Intermediates Electronic Materials Dicalcium Phosphate Company Profile

## Polymerization Catalysts

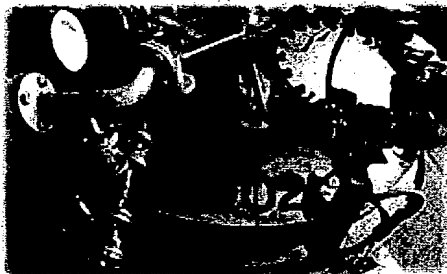
**Inquiry about this product:**

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- ▶ Ziegler-Natta catalyst
- ▶ Single-Site(Metallocene) catalyst

- **Single-Site (Metallocene) catalyst**

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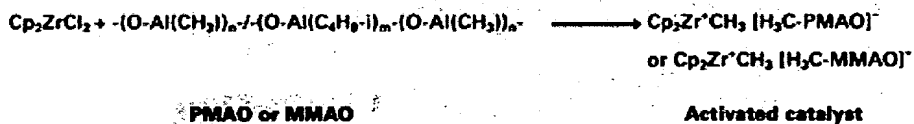


Tosoh Finechem boasts the most up-to-date polymerization catalyst production technology available. We have developed aluminum and boron type co-catalysts based on well-established alkyl aluminum and alkyl metal production technologies.

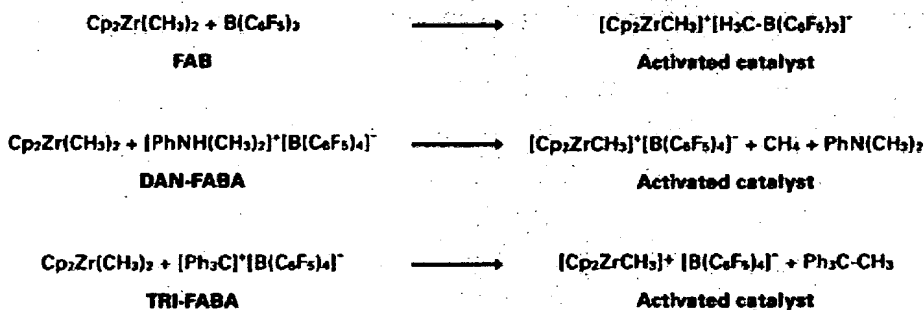
Our Ziegler-Natta catalyst production expertise makes supported metallocene catalysts tangible. Moreover, we provide supported metallocene catalysts as contract catalysts.

## Aluminoxane

**PMAO/MMAO(Polymethylaluminoxane/Modified methylaluminoxane**



## Borane/Borates





**BEST AVAILABLE COPY****Supported metallocene catalysts****Custom catalysts****Equipment**

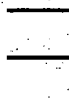
Support  
SiO<sub>2</sub>  
Co-catalyst  
MAO or Borane/borates



Supported co-catalysts

Reactor 200~6000L  
Pre-polymerization facility  
Dryer

Support  
SiO<sub>2</sub>  
Metallocene complex  
Co-catalyst  
MAO or Borane/Borates



Supported metallocene catalyst

● **Co-catalysts for metallocene polymerization catalysts system**

Type	Acronym	Chemical formula	Chemical name
Aluminum	PMAO	(O-Al(CH <sub>3</sub> )) <sub>n</sub> -	Methylaluminoxane
	TMAO-200 series	(O-Al(CH <sub>3</sub> )) <sub>n</sub> -	Methylaluminoxane (non-hydrolysis)
	MMAO-3A	(O-Al(CH <sub>3</sub> )) <sub>m</sub> -(O-Al(Bu-i)) <sub>n</sub>	Modified methylaluminoxane type 3
	TMAO-340 series	(O-Al(CH <sub>3</sub> )) <sub>m</sub> -(O-Al(Bu-i)) <sub>n</sub> -	Activated Modified methylaluminoxane
	PBAO	-(O-Al(C <sub>4</sub> H <sub>9</sub> -i)) <sub>n</sub> -	Polyisobutylaluminoxane
Borane	FAB/Isopar-E solution	B(C <sub>6</sub> F <sub>5</sub> ) <sub>3</sub>	Tris(pentafluorophenyl)borane
	DAN-FABA	[PhNH(CH <sub>3</sub> ) <sub>2</sub> ] <sup>+</sup> [B(C <sub>6</sub> F <sub>5</sub> ) <sub>4</sub> ] <sup>-</sup>	Dimethylanilinium tetrakis(pentafluorophenyl)borate
	Tri-FABA	[Ph <sub>3</sub> C] <sup>+</sup> [B(C <sub>6</sub> F <sub>5</sub> ) <sub>4</sub> ] <sup>-</sup>	Trityl tetrakis(pentafluorophenyl)borate
	Li-FABA	Li <sup>+</sup> [B(C <sub>6</sub> F <sub>5</sub> ) <sub>4</sub> ] <sup>-</sup>	Lithium tetrakis(pentafluorophenyl)borate

● **Supported metallocene co-catalysts and catalysts**

Type	Chemical formula	Remarks
Supported metallocene co-catalysts	MAO/SiO <sub>2</sub>	Custom catalysts
	Borane/SiO <sub>2</sub>	Custom catalysts
Supported metallocene catalysts	Metallocene/MAO/SiO <sub>2</sub>	Custom catalysts
	Metallocene/Borane/SiO <sub>2</sub>	Custom catalysts

► Polymerization Catalysts Top

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## Testing Method for Melt Flow Rate of Thermoplastics

### 1. Scope

This Japanese Industrial Standard specifies the testing method for the flow rate of thermoplastics for general application.

- Remarks 1. This method measures the rate of extrusion when molten thermoplastics are extruded through a die of specified length and diameter under specified temperature and pressure conditions.
2. This testing method includes Method A and Method B, Method A being a manual cutting-off method applicable to materials whose melt flow rates<sup>(1)</sup> are 0.1 to 25 g per 10 minutes and Method B being an automatic time measuring method applicable to materials whose melt flow rates<sup>(1)</sup> are 0.50 to 300 g per 10 minutes (refer to Remark of Table 3).

Note <sup>(1)</sup> This means the same as the melt flow index shown in JIS K 6900 and is expressed by the symbol MFR.

3. The melt flow properties of high polymers depend on the rate of shear. The rates of shear in this test are extremely lower than the rate of shear used in usual forming work, and therefore, precautions shall be taken because for many thermoplastics the data obtained by this method do not always correlate with the flow behavior in actual use of such plastics. However, this method is useful for quality control.
4. The units and numerical values shown in { } in this standard are based on the International System of Units (SI) and are appended for reference.

### 2. Definitions

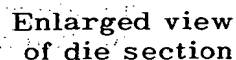
The main terms used in this standard shall be in accordance with JIS K 6900.

### 3. Apparatus and Appliances

3.1 Apparatus The apparatus used shall basically be an extrusion type plastometer which operates at a constant temperature and its general construction and dimensions are shown in Fig. 1. The sample is placed in a metal cylinder supported vertically and is extruded through a die by a weighted piston. The apparatus shall consist of the following main components:

Unit: mm.

## Plan

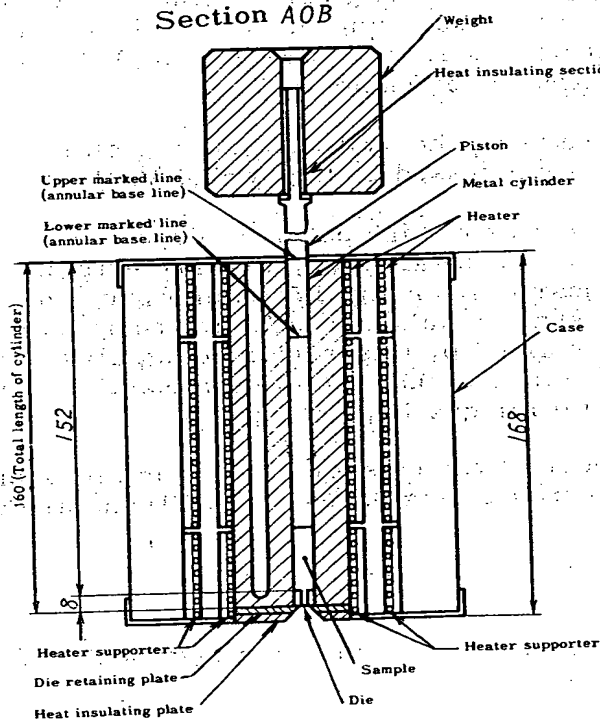


Technical drawing of a piston head assembly. The drawing shows a cross-section of the piston head, which is a cylindrical component with a central bore. The dimensions are as follows:

- Overall height:  $6.35 \pm 0.10$
- Top section height:  $8.00 \pm 0.025$
- Top section diameter:  $\phi 8.890 \pm 0.025$
- Middle section diameter:  $\phi 9.474 \pm 0.007$
- Bottom section diameter:  $\phi 9.550 \pm 0.007$
- Bottom section diameter:  $\phi 2.095 \pm 0.005$
- Bottom section diameter:  $\phi 9.50 \pm 0.03$

Labels in the drawing include:

- Piston head
- Sample
- Die



— 574 —

Note (2) It is recommended that the heat insulating plate be made of a material to which the extrudate does not stick, for example, a plate of polytetrafluoroethylene (approx. 3 mm in thickness).

3.1.2 Piston The piston<sup>(3)</sup> shall be made of metal, have a construction to allow a weight to be inserted in the upper portion, and have a heat insulating section to reduce the heat conduction from the piston to the weight. The piston head shall be  $9.474 \pm 0.007$  mm in diameter and  $6.35 \pm 0.10$  mm in length and the diameter of the portion of the piston above the piston head shall be decreased to approx. 9 mm. An upper marked line<sup>(4)</sup> shall be scribed shallowly on the piston axis at such a position that it agrees with the upper surface of the cylinder when the distance between the lower end of the piston head and the upper surface of the die is 20 mm, and in addition, a lower marked line at a position 30 mm below the upper marked line.

Notes (3) The piston and the cylinder shall be made of materials of different hardnesses. In this case, it is more convenient for maintenance, replacement, and the like to select the material of the cylinder to be harder than that of the piston.

Pistons for lighter loads shall preferably be hollow-structured. A suitable guide of little friction may also be provided to keep the piston vertical during measurement.

(4) Because the two marked lines show the limits for the working length of the cylinder, the cutting off of the sample shall be performed within these limits.

3.1.3 Heating and Temperature Adjusting Devices The heating and temperature adjusting devices used shall be capable of adjusting the temperature of the sample in the cylinder to within  $\pm 0.5^\circ\text{C}$  of the specified test temperature. The variation in temperature during the test shall be capable of being maintained within  $\pm 0.2^\circ\text{C}$ .

3.1.4 Thermometer for Apparatus The thermometer for apparatus, used for reading the temperature of the portion of the cylinder close to the die, shall be a mercury thermometer or a temperature detector which has been preliminarily calibrated so as to be capable of measuring temperatures with a precision of  $\pm 0.1^\circ\text{C}$ . A suitable low-melting alloy or silicone oil shall preferably be provided between the thermometer and the cylinder wall.

3.1.5 Die The die shall be made of steel and  $8.000 \pm 0.025$  mm in length and  $9.50 \pm 0.03$  mm in outer diameter. The inner diameter shall be  $2.095 \pm 0.005$  mm and the dimension shall be uniform lengthwise with a tolerance of  $\pm 0.005$  mm<sup>(5)</sup>.

Note (5) The surfaces of the die coming in contact with the sample during the test shall all be finished by lapping.

3.1.6 Weight The weight shall be used for being placed on the upper part of the piston to obtain the test load shown in Table 1. However, the test load referred to here shall include the mass of the piston, and its value shall be within a tolerance of  $\pm 0.5\%$ .

### 3.2 Appliances

3.2.1 Tool for Filling Sample in the Cylinder

3.2.2 Tool for Cutting Extruded Sample

3.2.3 Appliance for Cleaning

3.2.4 Stopwatch  $\pm 0.1$  s precision.

3.2.5 Balance  $\pm 0.5$  mg precision.

3.2.6 Calibration Thermometer Capable of measuring temperatures correctly to  $0.1^{\circ}\text{C}$ .

### 4. Sample

4.1 Form of Sample The sample used shall, as a rule, be in the form of pellets. However, it may also be in the form of powder, granules, aggregates or film, if the material can be packed in the cylinder bore. In the case of sample in the form of powder or the like, care shall be taken because air bubbles may be entrained in the extruded sample if the sample is preliminarily not be compressed.

4.2 Conditioning of Sample The sample shall be conditioned prior to the test according to the applicable material standards. If necessary, a stabilizer shall also be added. Materials containing volatile matter or hygroscopic materials shall be thoroughly dried preliminarily.

### 5. Test Conditions

5.1 As a rule, conditions suitable for the material shall be selected from the general test conditions<sup>(6)</sup> shown in Table 1. Test conditions generally used for respective resins are shown in Table 2 for reference.

Note <sup>(6)</sup> Where the application of the conditions shown in Table 1 are not suitable, other conditions may be used on agreement between the parties concerned.

Table 1. Test Conditions

Condition number	Test temperature (°C)	Test load (kgf){N}
1	125	0.325 { 3.19}
2	150	2.16 { 21.18}
3	190	0.325 { 3.10}
4	190	2.16 { 21.18}
5	190	5.00 { 49.03}
6	190	10.00 { 98.07}
7	190	21.60 {211.82}
8	200	5.00 { 49.03}
9	200	10.00 { 98.07}
10	210	10.00 { 98.07}
11	220	10.00 { 98.07}
12	230	0.325 { 3.19}
13	230	1.200 { 11.77}
14	230	2.16 { 21.18}
15	230	3.80 { 37.26}
16	230	5.00 { 49.03}
17	230	10.00 { 98.07}
18	275	0.325 { 3.19}
19	275	2.16 { 21.18}
20	280	2.16 { 21.18}
21	300	1.20 { 11.77}

Table 2. Generally Used Test Conditions for Respective Resins

Resin	Applicable conditions
Polyethylene	1, 3, 4, 5, 6, 7
Ethylene-vinyl acetate resin	1, 2, 3, 4
Polypropylene	4, 6, 14, 16, 17
Polyacetal	4
Cellulose acetate	4
Polystyrene	6, 8, 13, 15
ABS resin	8, 11, 15
Acrylic resin	9, 10, 13, 14, 15, 17
Polyamide	4, 12, 14, 18, 19
Polycarbonate	20, 21

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### 6. Temperature Calibration and Maintenance of Apparatus

6.1 Temperature Calibration The sample shall preliminarily be packed in the cylinder and when the temperature of the thermometer for apparatus has reached the test temperature, the temperature shall be measured by directly inserting the calibration thermometer into the sample at a position about 10 mm above the upper surface of the die. The reading of the thermometer for apparatus shall be calibrated by using the difference between the reading of the calibration thermometer and that of the thermometer for apparatus, and then the temperature of the cylinder shall be adjusted so that the calibrated reading agrees with the test temperature.

6.2 Cleaning of Apparatus The test apparatus shall be thoroughly cleaned after each measurement. The cylinder and piston shall be cleaned by wiping with a cloth and the die, by putting through it a brass or wooden rod. If required, they may be cleaned with a solvent. Alternatively, burning in a nitrogen atmosphere of approx. 550 °C may also be used. Materials or abrasives likely to damage the piston, cylinder or die, may not be used. Where solvents at a high temperature are used, special care shall be taken to protect the test operators from exposure to toxic vapors.

6.3 Cleaning of Lower Portion of Cylinder In an apparatus in constant use, as required, the cylinder shall be thoroughly cleaned by detaching the heat insulating plate and die retaining plate.

6.4 Alignment of Cylinder It shall be insured, before using the apparatus, that the cylinder is correctly positioned vertical.

6.5 The dimensional range of the diameters of the sample inserting hole, piston head and die shall be confirmed periodically by using a plug gauge, limit gauge, or the like.

### 7. Procedure — Method A (Manual Cutting-Off Method)

The following procedure shall be performed:

7.1 After cleaning the apparatus, maintain it at the specified temperature at least for 15 minutes with the piston and die attached. However, where tests are successively carried out, it is not necessary to maintain the specified temperature for 15 minutes in the second and succeeding tests.

7.2 After preliminarily confirming the estimated MFR value of the sample, place the sample of the charge weight specified in Table 3 in the cylinder, put on the piston, and force it into the cylinder by hand. This charging operation shall be completed within one minute to prevent the degradation of the sample. Taking the moment when the charging has been completed as the starting point of preheating, start time measurement at this moment (7). Although it depends on the sample whether or not a weight is to be put on the piston, place the piston in the cylinder in all cases.

Table 3

MFR (g/10 min)	Charge weight (g)	Time for sampling (s)
0.1 to 0.5	3 to 5	240
0.5 to 1.0	3 to 5	120
1.0 to 3.5	3 to 5	60
3.5 to 10	5 to 8	30
10 to 25	5 to 8	5 to 15

**Remark:** The respective results of measurement may be taken as the MFR values, under the conditions of the division of 10 to 25 for the sample having MFR > 25, and under the conditions of the division of 0.1 to 0.5 for the sample having MFR < 0.1. For sample having MFR values of 0.5, 1.0, 3.5 and 10, the next longer sampling time shall be adopted.

**Note (7)** The cylinder temperature shall have been restored to the specified temperature within 4 minutes of starting the time measurement.

7.3 Discard the extrudate 6 minutes after preheating. At this moment, the lower marked line of the piston shall be positioned directly beneath the upper surface of the cylinder<sup>(8)</sup>. Cut off the extrudate pushed out thereafter when the time shown in Table 3 has been reached, and using the extrudate as the sample to be weighed, provided that it is free from air bubbles, weigh it accurately to 1 mg after cooling.

**Note (8)** For sample having large MFR values, the piston rod may be used singly, or with a light weight placed on it, for the earlier period of preheating to prevent premature flowing out of the sample during preheating. However, the load shall be changed to the normal load after 5 minutes at least.

If necessary, a piston retaining rod may be used. The piston retaining rod, used for retaining the test load to prevent the sample from flowing out during the preheating period, shall consist of a material having thermal resistance and a low thermal conductivity (for example, phenol resin), be capable of retaining the piston at the position where the lower marked line of the piston is 15 mm above the upper surface of the cylinder, and have a construction capable of being readily detached. If such a piston retaining rod is used, the normal load shall be applied from the beginning. The piston retaining rod shall be detached after 5 minutes and the piston shall be lowered under the self-weight of the piston and the weight.

For sample having small MFR value, if necessary, the piston may be pushed down manually or by other means in such a manner that 5 minutes after starting preheating, the lower marked line of the piston is positioned 0 to 10 mm above the upper surface of the cylinder.



7.4 As a rule, the test shall be performed by taking one sample to be weighed with each extruding operation for the same material and repeating this operation 3 times, but alternatively, 3 samples to be weighed may be taken continuously during one extruding operation<sup>(9)</sup>. A retest shall be performed if the difference between the largest and smallest value of the individual measured values exceeds 15 % of the mean value or if the extrudate contains air bubbles.

The sample shall be cut off between the two marked lines of the piston.

Note <sup>(9)</sup> It shall be decided on agreement between the parties concerned which of these methods is to be adopted.

7.5 Expression of Results MFR shall be calculated as the mass (g) of the sample extruded in 10 minutes from the following formula:

$$\text{MFR}(T, M, A) = \frac{600 \times m}{t}$$

where

$T$  : measured temperature (°C)

$M$  : test load (kgf) { N }

$A$  : symbol indicating Method A

$m$  : mean mass value of cut-off sample (g)

$t$  : time for taking sample to be weighed (s)

600 : number of seconds for 10 minutes

The test results shall be reported by rounding the arithmetical mean of the results of 3 measurements to two significant figures in accordance with JIS Z 8401.

## 8. Procedure — Method B (Automatic Time Measuring Method)

### 8.1 Apparatus

8.1.1 The apparatus shall consist of an extrusion type plastometer and appliances as mentioned in 3. , in addition to the following component:

8.1.2 Automatic Time Measuring Device This device shall be a device which measures electrically or mechanically the time in which the piston moves by the specified distance in the plastometer, and shall consist of a timer-actuating switch.

The timer-actuating switch shall actuate a timer in response to a piston displacement of 4.0 mm for measuring MFR values of 0.50 to 10, and in response to a piston displacement of 25.0 mm for measuring MFR value of 10 to 300.

The timer shall be capable of reading to 0.1 second.

### 8.2 Procedure

8.2.1 The procedures other than the following shall be performed as in 7.

8.2.2. Setting of Timer-Actuating Switch The timer-actuating switch shall be set so that the piston operates at the specified portion of the cylinder. The setting of the timer-actuating switch shall be decided so that first, the timer starts operation when the lower end of the piston head comes to a position  $50 \pm 1.5$  mm above the upper surface of the die and next, the timer stops when the piston head is positioned  $4.0 \pm 0.25$  mm below the said position for measuring MFR values of 0.50 to 10, and  $25.0 \pm 0.25$  mm below the said position for measuring MFR values of 10 to 300. Adjustment shall be made so that the time of piston movement is indicated or recorded. In both cases, the moving distance shall be checked with a precision of  $\pm 0.025$  mm.

8.2.3 The sample shall be charged in the cylinder in accordance with Table 4 according to the anticipated MFR value.

Table 4

MFR (g/10 minutes)	Charge weight (g)
0.5 to 3.5	3 to 5
3.5 to 300	5 to 8

8.2.4 The procedures to be carried out from charging of the sample in the cylinder to the start of measurement shall be in accordance with 7. However, adjustment shall be made so that the timer actuates after the total preheating time of between 6 and 8 minutes.

8.2.5 The time in which the piston moves over the checked distance shall be measured to 0.1 sec.

8.2.6 The test shall be repeated 3 times. A retest shall be performed when the difference between the maximum and the minimum values has exceeded 15 % of the mean value or when the extrudate contains air bubbles.

8.3 Expression of Results MFR shall be calculated as the mass (g) of the sample extruded in 10 minutes from the following formula:

$$\text{MFR}(T, M, B) = \frac{426 \times L \times \rho}{t}$$

where  $T$ : measured temperature ( $^{\circ}\text{C}$ )

$M$ : test load (kgf) { N }

$B$ : symbol indicating Method B

$L$ : moving distance of piston (cm)

$\rho$ : density of resin at the test temperature ( $\text{g}/\text{cm}^3$ ) (10)

$t$ : mean value of time required for piston to move length  $L$  (s)

426 : in the case where the test apparatus is used at it stands, [ mean value of piston and cylinder areas ( $\text{cm}^2$ ) ]  $\times$  600 (number of seconds for 10 minutes)

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The test results shall be expressed in two significant figures by rounding the arithmetical mean of the results of 3 measurements according to JIS Z 8401.

Note (10) The density  $\rho$  ( $\text{g}/\text{cm}^3$ ) of resin at the test temperature varies with the kind of resin and the test temperature. Although suitable published values, if available, may be utilized, it is recommended that actual measurement be made as follows: When the piston moves by 25.0 mm,  $1.776 \text{ cm}^3$  of resin is extruded, so that if the mass  $W$  (g) of this extrudate is measured,  $\rho$  is calculated from the formula  $\rho = W/1.776$ .

## 9. Report

The report shall contain information corresponding to the following items:

- (1) Kind of material tested
- (2) Form of sample
- (3) Conditions used for conditioning of sample
- (4) Test temperature and test-load
- (5) Test method (Method A or Method B), and in the case of Method A, the method of cutting off the sample (refer to 7.4)
- (6) Melt flow rate (MFR)
- (7) Abnormal behaviors of sample, such as discoloration, sticking, distortion of extrudate, and abnormal variation in melt flow rate (MFR)
- (8) Atmospheric conditions of testing room
- (9) Date of test
- (10) Matters decided on agreement between the parties concerned

## Flow Testing Method (Reference Test)

### 1. Scope

This standard specifies the testing method for the flow value of thermoplastics under heavy loads.

Remark: This method measures the flow values under heavy load with high rate of shearing used during normal forming work. However, this method is shown here as a reference test method because it is still not so frequently put to general use.

### 2. Apparatus and Appliances

#### 2.1 Apparatus

2.1.1 A flow testing machine as shown in Reference Fig. 1 shall be used for measuring the flow value.

The flow testing machine shall consist of a heating unit for heating the sample, a piston capable of applying a specified pressure to the sample, and a die through which the sample is made flow out.

2.1.2 The heating unit shall be provided with a cylinder, die inserting section, heat generating element, and temperature adjusting device for adjusting the heating temperature.

2.1.3 The cylinder shall be a metal cylinder of 60 mm in outer diameter and 71 mm in length and be provided with a sample introducing hole of  $11.329 + 0.005_0$  mm in diameter and 41 mm in length, and holes for inserting a thermometer and a heat sensitive element for temperature adjustment, with the bottom of the sample inserting hole fitted with a retainer for retaining a die.

2.1.4 The die inserted in the bottom of the sample introducing hole of the cylinder shall be made of metal and its dimensions and shape shall, as a rule, be as shown in Reference Figs. 3 and 4 and have surfaces finished by lapping.

2.1.5 The piston shall be made of metal and have a total length of 45 mm, with the head being  $11.282 + 0.002_0$  mm in diameter and 20 mm in length and finished by lapping. The diameter of the portion above the piston head shall be 10 mm.

2.1.6 The pressure applied to the piston shall be 10 to 500 kgf/cm<sup>2</sup> { 0.98 to 49.03 MPa } and the specified precision of the test load shall be  $\pm 1\%$  of the specified value.

2.1.7 The heating unit shall have a construction capable of maintaining the cylinder at the temperature required for the test in its areas within 10 mm of the upper surface of the die by a suitable means of heating.

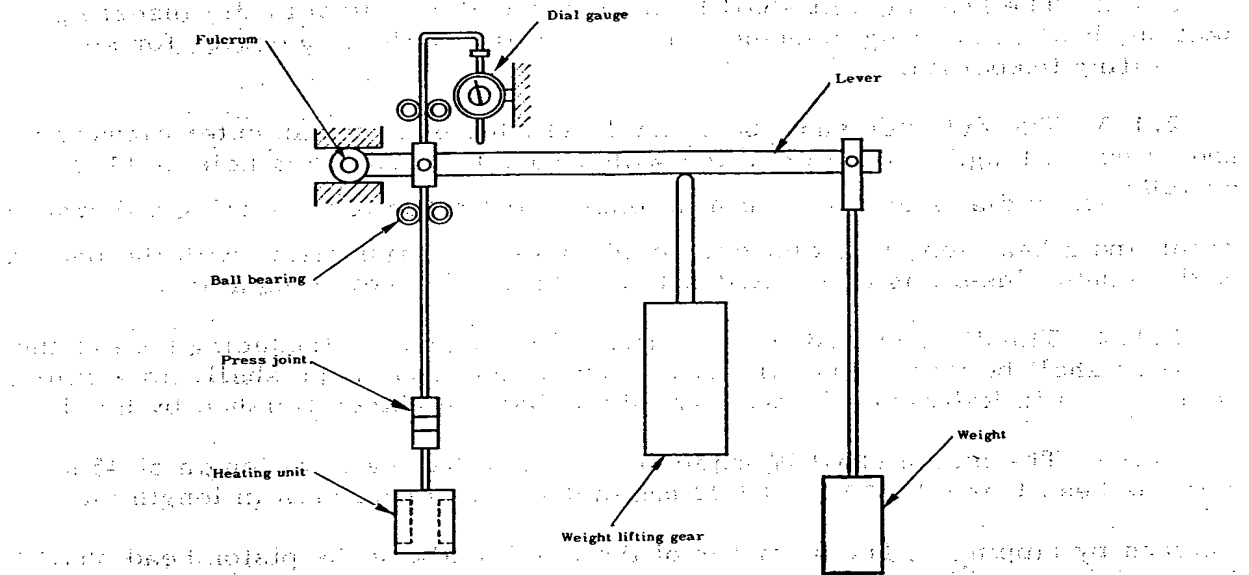
2.1.8 The temperature adjusting device of the heating unit shall be capable of maintaining a constant temperature within a tolerance of  $\pm 0.5^\circ\text{C}$  in the range of temperature of 80 to 320  $^\circ\text{C}$ .

2.1.9 The scale of the thermometer for the apparatus shall be capable of reading to  $0.5^\circ\text{C}$  and be preliminarily calibrated to the test temperature in the following manner:

When the sample is filled in the cylinder and the temperature indicated by the thermometer for apparatus has reached the test temperature, a calibration thermometer shall be directly inserted in the sample and the temperature of the sample measured. The reading of the thermometer for apparatus shall be calibrated from the difference between the reading of the calibration thermometer and that of the thermometer for apparatus, and the temperature of the cylinder shall be adjusted so that the calibrated reading agrees with the test temperature.

2.1.10 The apparatus shall be provided with a device which records the quantity of lowering of the piston. This recording device shall be capable of recording the quantity of the sample which has passed through the die (the quantity of lowering of the piston of a given sectional area) as a function of time.

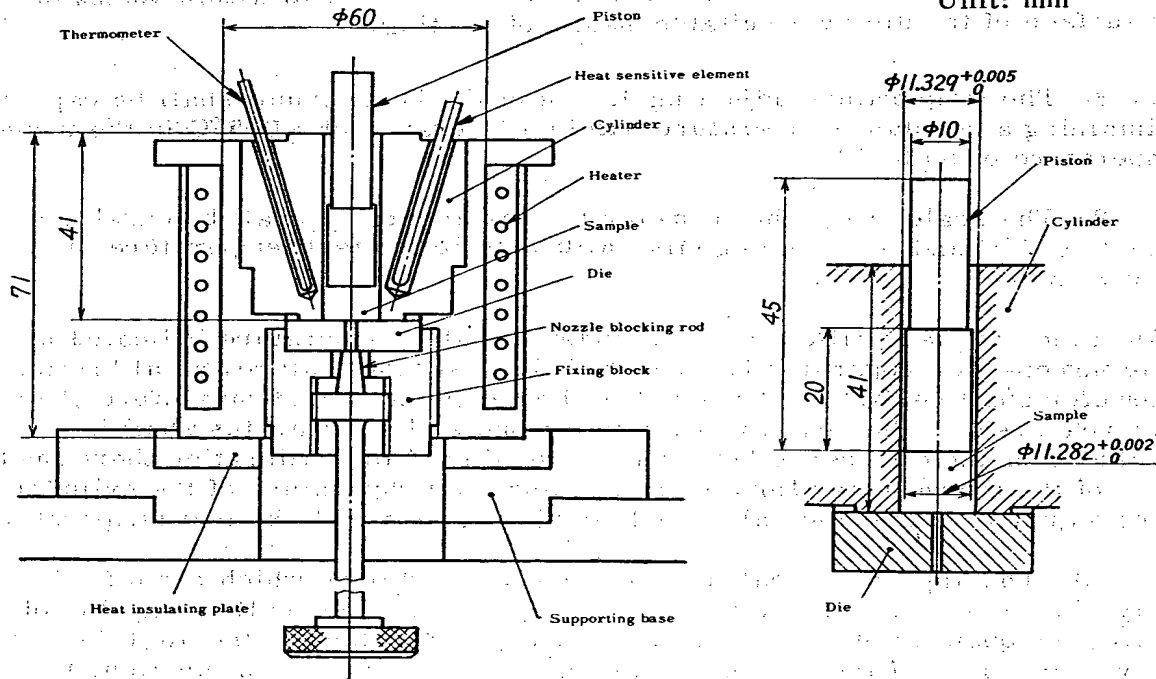
Reference Fig. 1. Flow Testing Machine



Reference Fig. 2. Test Apparatus

Enlarged View of Die Section

Unit: mm

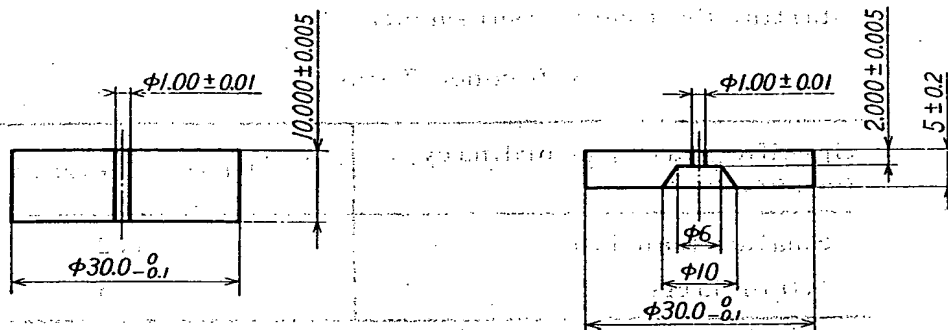


Reference Fig. 3. Die A

Reference Fig. 4. Die B

Unit: mm

Unit: mm



## 2.2 Appliances

### 2.2.1 Tool for filling sample in the cylinder.

### 2.2.2 Appliance for cleaning.

## 3. Sample

The sample taken according to the melt flow rate testing method specified in the applicable standard.

## 4. Test Conditions

The test temperature, test load and the die shall be decided according to the applicable standard or on agreement between the parties concerned.

## 5. Procedures

5.1 Procedures without Gas Removal Operation The following procedures shall be performed:

5.1.1 After cleaning<sup>(1)</sup> the apparatus, maintain the specified temperature for at least 2 minutes with the piston and die attached in place.

Note <sup>(1)</sup> The procedure of cleaning shall be in accordance with the melt flow rate testing method.

5.1.2 Place the sample of the charge weight specified in Reference Table 1 in the piston<sup>(2)</sup> and firmly tighten the press joint. This packing operation shall be completed within one minute to prevent the degradation of the sample. Take the moment the filling operation is finished as the starting point of preheating and start time measurement at this point<sup>(3)</sup>.

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- Notes (2) While inserting the sample, the piston shall be kept on the heating unit to prevent cooling.
- (3) The temperature shall have been restored within 4 minutes from starting the time measurement.

Reference Table 1

Specific gravity at ordinary temperature	Charge weight (g)
Smaller than 1.0	1.2
1.0 or larger	1.5

5.1.3 Five minutes after starting preheating, apply the test load via the piston and draw a flow curve by taking the lowering quantity of the piston as a function of the flowing out time<sup>(4)</sup>.

Note (4) The recording device shall be started about 10 seconds earlier than the application of the test load. As a rule, the recording device shall be so operated that the flow curve shows a gradient of about 45 degrees when the lowering quantity of the piston is 3 to 7 mm.

5.2 Procedures with Gas Removing Operation The following procedures shall be performed:

5.2.1 The same operation as in 5.1.1 shall be performed. However, in this case a nozzle blocking rod shall be attached.

5.2.2 Place the sample of the charge weight specified in Reference Table 1 in the cylinder<sup>(2)</sup>. This filling operation shall be completed within one minute to prevent the degradation of the sample. Taking the moment the filling operation is finished as the starting point of preheating, start time measurement at this point<sup>(3)</sup>.

5.2.3 Three minutes after starting preheating, insert the piston into the cylinder and after repeating the application of the test load and the removal of it 3 or 4 times<sup>(5)</sup>, let it stand with the load removed<sup>(6)</sup>.

Notes (5) This operation shall be performed within 1 minute.

(6) The press joint shall be kept in slight contact with the piston.

5.2.4 Five minutes and 30 seconds after the start of preheating, remove the nozzle blocking rod.

5.2.5 Six minutes after the start of preheating, perform the same operation as in 5.1.3.

5.3 The test shall be repeated 3 times as a rule.

6. Expression of Test Results

The flow value  $Q$  shall be calculated from the following formula and expressed in two significant figures by rounding off according to JIS Z 8401.

$$Q(T, M, D_a, D_i) = \frac{0.4}{t}$$

where

$T$  : measured temperature (°C)

$M$  : test load (kgf) { N }

$D_a$  : diameter of die (mm)

$D_i$  : length of die (mm)

$t$  : mean value of the time required for the piston to lower from 3 mm to 7 mm after the start of flowing out under the test load (s)

7. Report

The report shall contain information corresponding to the following items:

- (1) Kind of material tested
- (2) Form of sample
- (3) Conditions used for conditioning of sample
- (4) Test temperature and test load
- (5) Material quality, diameter and length of die used for test
- (6) Flow value (ml/sec)
- (7) Abnormal behaviors of sample, such as discoloration, sticking, distortion of extrudate, and abnormal flow curve
- (8) Atmospheric conditions of testing room
- (9) Date of test
- (10) Other particulars agreed on between the parties concerned





Designation: D 1238 - 04c

# Standard Test Method for Melt Flow Rates of Thermoplastics by Extrusion Plastometer<sup>1</sup>

This standard is issued under the fixed designation D 1238; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## Scope\*

1.1 This test method covers measurement of the rate of extrusion of molten resins through a die of a specified length and diameter under prescribed conditions of temperature, load, and piston position in the barrel as the timed measurement is being made.

1.2 Procedure A is a manual cutoff operation based on time used for materials having flow rates that fall generally between .15 and 50 g/10 min. Procedure B is an automatically timed flow rate measurement used for materials having flows from .50 to 900 g/10 min. By both procedures, the piston travel is generally the same during the timed measurement; the piston travel is about 46 and 20.6 mm above the die. Comparable flow rates have been obtained by these procedures in interlaboratory round-robin measurements of several materials described in 4.1. Provision is made for calculation of melt volume-flow rate as well as melt mass-flow rate.

NOTE 1—Round-robin testing indicates this test method may be suitable at flow rates up to 1500 g/10 min if the timing clock resolves the elapsed time to the nearest 0.01 s.

NOTE 2—This test method and ISO 1133-1991 are technically equivalent.

1.3 This standard does not purport to address the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Specific precautionary statements are given in 5.7, 10.2.12, and 15.1.2.

## Referenced Documents

### 2.1 ASTM Standards:<sup>2</sup>

- D 618 Practice for Conditioning Plastics for Testing
- D 883 Terminology Relating to Plastics
- D 3364 Test Method for Flow Rates for Poly Vinyl Chloride

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.30 on Thermal Properties of Plastics (D20.30.08).

Current edition approved December 1, 2004. Published December 2004. Originally approved in 1965. Last previous edition approved in 2004 as D 1238 - 04b.

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or use the ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For Annual Book of ASTM Standards volume information, refer to the standard's Document Summary page on the ASTM website.

with Molecular Structural Implications

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

### 2.2 ANSI Standard:

B46.1 on Surface Texture<sup>3</sup>

### 2.3 ISO Standard:

ISO 1133-1991 Determination of the Melt-Mass Flow Rate (MFR) and the Melt Volume-Flow Rate (MVR) of Thermoplastics<sup>3</sup>

## 3. Terminology

### 3.1 General:

3.1.1 For definition of some of the technical terms used in this test method refer to Terminology D 883.

## 4. Significance and Use

4.1 This test method is particularly useful for quality control tests on thermoplastics.

NOTE 3—Polymers having flow rates less than 0.15 or greater than 900 g/10 min may be tested by the procedures in this test method; however, precision data have not been developed.

4.2 This test method serves to indicate the uniformity of the flow rate of the polymer as made by an individual process and, in this case, may be indicative of uniformity of other properties. However, uniformity of flow rate among various polymers as made by various processes does not, in the absence of other tests, indicate uniformity of other properties.

4.3 The flow rate obtained with the extrusion plastometer is not a fundamental polymer property. It is an empirically defined parameter critically influenced by the physical properties and molecular structure of the polymer and the conditions of measurement. The rheological characteristics of polymer melts depend on a number of variables. Since the values of these variables occurring in this test may differ substantially from those in large-scale processes, test results may not correlate directly with processing behavior.

4.4 The flow rate of a material may be measured under any of the conditions listed for it in 8.2. Additional characterization of a material can be obtained if more than one condition is

<sup>3</sup> Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

\*A Summary of Changes section appears at the end of this standard.

used. In case two conditions are employed, a Flow Rate Ratio (FRR) may be obtained by dividing the flow rate at one condition by the flow rate at the other condition.

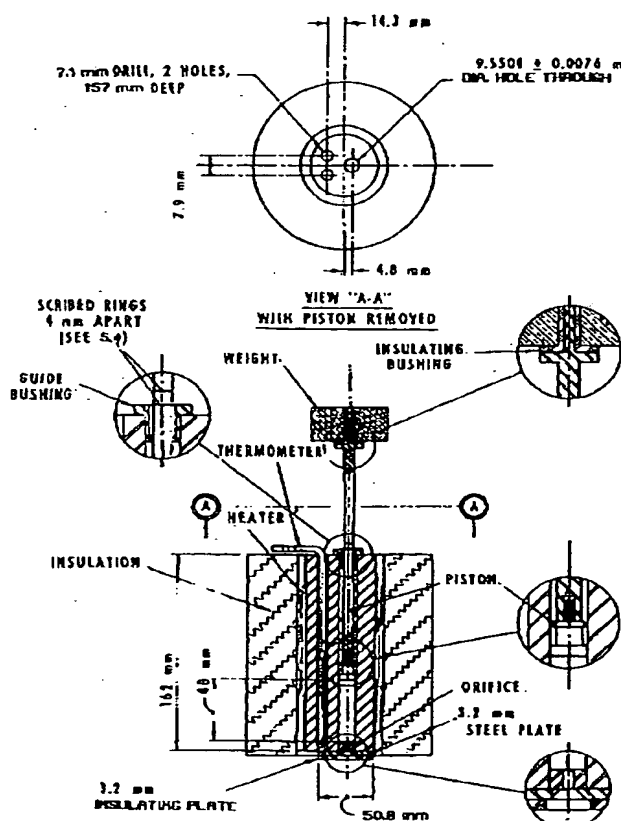
## 5. Apparatus

### 5.1 Plasiometer.

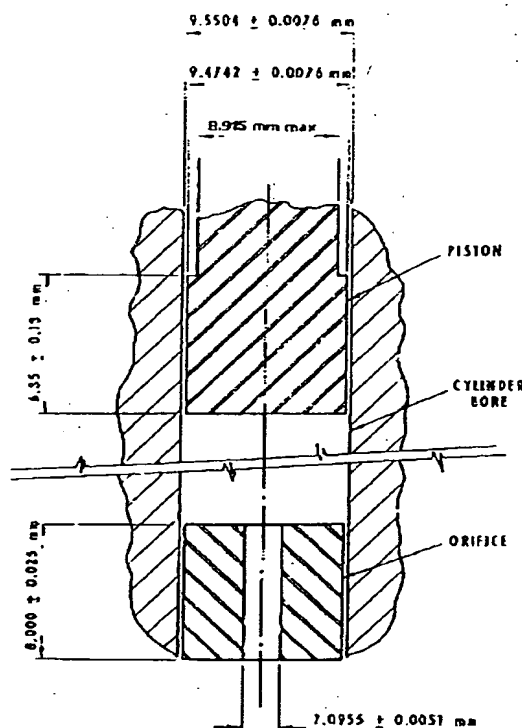
5.1.1 The apparatus shall be a dead-weight piston plastometer consisting of a thermostatically controlled heated steel cylinder with a die at the lower end and a weighted piston operating within the cylinder. The essential features of the plastometer, illustrated in Figs. 1 and 2, are described in 5.2-5.8. All dimensional measurements shall be made when the article being measured is at  $23 \pm 5^\circ\text{C}$ .

5.1.2 Relatively minor changes in the design and arrangement of the component parts have been shown to cause differences in results among laboratories. It is important, therefore, for the best interlaboratory agreement that the design adhere closely to the description herein; otherwise, it should be determined that modifications do not influence the results.

**5.2 Cylinder**—The steel cylinder shall be 50.8 mm in diameter, 162 mm in length with a smooth, straight hole  $9.5504 \pm 0.0076$  mm in diameter, displaced 4.8 mm from the cylinder axis. Wells for a thermal sensor (thermoregulator, thermistor, etc.) and thermometer shall be provided as shown



**FIG. 1 General Arrangement of Extrusion Plastometer**



**FIG. 2 Details of Extrusion Plastometer**

in Fig. 1. A 3.2-mm plate shall be attached to the bottom of the cylinder to retain the die. A hole in this plate, centered under the die and countersunk from below, allows free passage of the extrudate. The cylinder may be supported by at least two 6.4-mm high-strength screws at the top (radially positioned at right angles to the applied load) or by at least two 10-mm diameter rods screwed into the side of the cylinder for attaching to a vertical support. The essential dimensions of a satisfactory cylinder of this type are shown in Fig. 1 (Note 4). The cylinder bore should be finished by techniques known to produce approximately 12 rms or better in accordance with ANSI B46.1.

NOTE 4—Cylinders made of SAE 52100 or other equivalent steel heat-hardened to 60-65 Rockwell Hardness Scale C give good service when used at temperatures below 200°C. Cylinder liners of cobalt-chromium-tungsten alloy are also satisfactory to 300°C.

**5.3 Die**—The outside of the steel die shall be such diameter that it will fall freely to the bottom of the  $9.5504 \pm 0.0076$  mm diameter hole in the cylinder (Note 5). The die shall have a smooth straight bore  $2.0955 \pm 0.0051$  mm in diameter and shall be  $8.000 \pm 0.025$  mm in length. The bore and its finish are critical. It shall have no visible drill or other tool marks and no detectable eccentricity. The die bore shall be finished by techniques known to produce approximately 12 rms or better in accordance with ANSI B46.1.

NOTE 5—Recommended die material is tungsten carbide. Also satisfactory are steel, synthetic sapphire, and cobalt-chromium-tungsten alloy.

#### 5.4 Piston:

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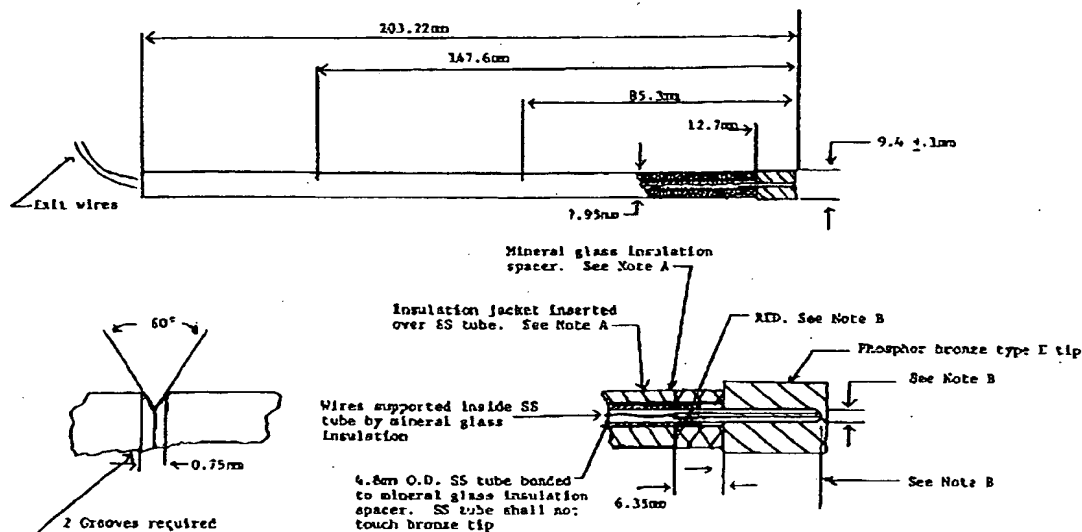


FIG. 3 Example of a Temperature Calibration Device

Note A—Mineral glass insulation or equivalent spacer shall be bonded to tip and SS tube. Bond material shall be low conductivity type, 400°C minimum rating. Insulation jacket material shall be low conductivity type (400°C minimum rating preferred, see Note 5).

Note B—The RTD shall be inserted into bronze tip and bonded using high conductivity, 400°C rated material. Tip of RTD element shall touch the bronze tip. Minimum insertion depth of 11.2 mm clearance between RTD and tip wall shall be minimized.

5.4.1 The piston shall be made of steel with an insulating bushing at the top as a barrier to heat transfer from the piston to the weight. The land of the piston shall be  $9.4742 \pm 0.0076$  mm in diameter and  $6.35 \pm 0.13$  mm in length. The piston design may incorporate means for land replacement, for example, having threads and flats immediately above the land. Above the land, the piston shall be no larger than 8.915 mm in diameter (Note 6). The finish of the piston foot shall be 12 rms in accordance with ANSI B46.1. If wear or corrosion is a problem, the piston should be of stainless steel and equipped with a detachable foot for ease of replacement.

Note 6—To improve standardization it is preferable that the piston be guided with a loose-fitting metal sleeve at the top of the cylinder.

Note 7—Pistons of SAE 52100 steel with the bottom 25 mm, including the foot, hardened to a Rockwell hardness, C scale, of 55 to 59 have been found to give good service when used at temperatures below 200°C.

5.4.2 The piston shall be scribed with two reference marks 4 mm apart in such fashion that when the lower mark coincides with the top of the cylinder or other suitable reference point, the bottom of the piston is 48 mm above the top of the die (see Fig. 1).

5.4.3 The combined weight of piston and load shall be within a tolerance of  $\pm 0.5$  % of the selected load.

#### 5.5 Heater:

5.5.1 The equipment must have a heater capable of heating the apparatus so that the temperature at 10 mm above the die can be maintained within  $\pm 0.2^\circ\text{C}$  of the desired temperature during the test. The temperature of the barrel, from 10 mm to 75 mm above the top of the die, must be maintained within  $\pm 1$  % of the set temperature ( $^\circ\text{C}$ ).

Note 8—At temperatures higher than 200°C this degree of temperature control may be more difficult to obtain.

5.5.2 Calibrate the temperature-indicating device by means of a light-gage probe-type thermocouple or a platinum-resistance temperature sensor having a short sensing length.<sup>4</sup> The thermocouple should be encased in a metallic sheath having a diameter of approximately 1.6 mm with its hot junction grounded to the end of the sheath. Insert the temperature sensor into the melt from the top of the cylinder so that it is  $10 \pm 1$  mm above the upper face of the die. The temperature sensors shall be used with a potentiometer having a sensitivity of at least 0.005 mV, or a temperature readout having a sensitivity of at least  $0.1^\circ\text{C}$ . Calibration should also be verified at 75 mm above the upper face of the die. An alternate technique for calibration is to use a sheathed thermocouple or platinum-resistance temperature sensor with tip diameter of  $9.4 \pm 0.1$  mm for insertion in the bore without material present. An example of this is shown in Fig. 3. Calibration of the temperature-indicating device shall be verified at each run temperature.

Note 9—The response of the temperature sensing device may be affected by immersion level. Take care to ensure adequate insulation of the device sensor and stabilization of the barrel temperature.

5.5.3 Heat shall be supplied by electric band heater(s) covering the entire length of the cylinder. The heater(s) may be

<sup>4</sup> Round-robin data showing flow rate and corresponding temperature profile of the melt obtained using probe-type thermocouples and platinum resistance temperature sensors can be obtained from ASTM Headquarters. Request RR:D20-1094.

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single- or multi-element, depending upon the manufacturer's control means. The heater(s) plus control system must be capable of maintaining the temperature within the required  $\pm 0.2^\circ\text{C}$  of the set point. The temperature sensor and readout equipment must be calibrated to a traceable national standard (that is, NIST) at least once per year. The cylinder with the heater(s) shall be lagged with 38 mm of foamed-glass insulation. An insulating plate 3.2 mm in thickness shall be attached to the bottom of the cylinder to minimize heat loss at this point.

**5.6 Temperature Controller**—The type of controller and sensor must be capable of meeting the required control tolerance specified in 5.5.1.

**5.7 Thermometer**—Thermometers having a range of  $4^\circ\text{C}$  graduated in  $0.2^\circ\text{C}$  divisions may be used to indicate temperature. The temperature at this point may not necessarily be the temperature of the material 10 mm above the die. The thermometer may be used to monitor indirectly the temperature of the material 10 mm above the die and may be calibrated by reference to a thermocouple or platinum resistance temperature sensor inserted in the material 10 mm above the die. See 5.5.2 for a description of a method for measuring temperature. (Warning—Caution should be observed with the use of a mercury-filled thermometer. Mercury vaporization occurs if the thermometer is broken. Mercury thermometers are not to be used at or above the boiling point of mercury, which is  $357^\circ\text{C}$ .)

**5.8 Level**—Provision shall be made for vertical alignment of the bore of the extrusion plastometer. This is necessary to minimize subtractive loads resulting from rubbing or friction between the piston tip and sidewall. Means of alignment are discussed in Appendix X1.

**5.9 Accessory Equipment**—Necessary accessories include equipment for charging samples to the cylinder, a funnel, a die plug, a tool for cutting off the extruded sample, a timer or stop watch, cleaning equipment, go/no-go gages, a balance accurate to  $\pm 0.001$  g, and, when required, a weight or weight-piston support.

**Note 10**—Satisfactory operation of the apparatus for polyethylenes can be ascertained by making measurements on NIST Standard Reference Materials (SRMs) certified for melt flow rate. The four SRMs certified under condition 190/2.16 are SRM 1473 with a flow rate of 1.29 g/min, SRM 1474 with a flow rate of 5.03 g/10 min, SRM 1496 with a flow rate of 0.26 g/10 min, and SRM 1497 with a flow rate of 0.19 g/10 min. SRM 1475a is certified under condition 190/3.25 with a flow rate of 2.20 g/10 min.<sup>3</sup>

## 6. Test Specimen

6.1 The test specimen may be in any form that can be introduced into the bore of the cylinder, for example, powder, granules, strips of film, or molded slugs. It may be desirable to preform or pelletize a powder.

## 7. Conditioning

7.1 Many thermoplastic materials do not require conditioning prior to testing. Materials which contain volatile components, are chemically reactive, or have other special character-

istics most probably require appropriate conditioning procedures. Moisture not only affects reproducibility of rate measurement but, in some types of materials, degradation is accelerated by moisture at the high temperatures used in testing. Check the applicable material specification for conditioning requirements before using this test. See Pr. D 618 for appropriate conditioning practices.

## 8. Procedural Conditions

8.1 Standard conditions of test are given in Table 1 conditions shall be shown as: Condition \_\_\_\_ / \_\_\_\_, where temperature in degrees Celsius is shown first, followed by weight in kilograms. For example: Condition 190/2.16.

8.2 The following conditions have been found satisfactory for the material listed:

Material	Condition	
Acetals (copolymer and homopolymers)	190/2.16	190/
Acrylics	230/1.2	230/
Acrylonitrile-butadiene-styrene	200/5.0	230/
	220/10	
Acrylonitrile-butadiene-styrene/polycarbonate blends	230/3.8	250/
	265/3.8	265/
Cellulose esters	190/0.325	190/
	190/21.60	210/
Ethylene-chlorotrifluoroethylene copolymer	271.5/2.16	
Ethylene-tetrafluoroethylene copolymer	297/5.0	
Nylon	275/0.325	235/
	235/2.16	235/
	275/5.0	
Perfluoro(ethylene-propylene) copolymer	372/2.16	
Perfluoroalkoxyalkane	372/5.0	
Polycaprolactone	125/2.16	80/2
Polychlorotrifluoroethylene	265/12.5	
Polyether sulfone (PES)	380/2.16	360/
	343/2.16	
Polyethylene	125/0.325	125/
	250/1.2	
	190/0.325	190/
	190/21.60	190/
	310/12.5	
Polycarbonate	300/1.2	
Polymono-chlorotrifluoroethylene	265/21.6	
	265/31.6	
Polypropylene	230/2.16	
Polyphenyl sulfone (PPSU)	365/5.0	380/
Polystyrene	200/5.0	230/
	230/3.8	190/
Polysulfone (PSU)	343/2.16	360/
Polyterephthalate	250/2.16	210/
	285/2.16	
Poly(vinyl acetate)	150/21.6	
Poly(vinylidene fluoride)	230/21.6	
	230/5.0	
Poly(phenylene sulfide)	315/5.0	
Styrene acrylonitrile	220/10	230/
	230/3.8	
Styrenic Thermoplastic Elastomer	190/2.16	200/
Thermoplastic Elastomer-Ether-Ester	190/2.16	220/
	230/2.16	240/
		250/
Thermoplastic elastomers (TEO)	230/2.16	
Vinylidene fluoride copolymers	230/21.6	
	230/5.0	

for  $T_m = 100^\circ$  use 120/5.0 or

**Note 11**—Some materials may require special materials of construction or handling for performing this test. Please refer to the material specification for appropriate recommendations.

8.3 If more than one condition is used and the material is polyethylene, the determination of Flow Rate Ratio (FRR) has been found to be useful. The FRR is a dimensionless number

<sup>3</sup> These standard polyethylenes are available from the National Institute of Standards and Technology, Office of Standard Reference Materials, Washington, DC 20234.



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TABLE 1 Standard Test Conditions, Temperature, and Load

Condition Standard Designation	Temperature, °C	Total Load Including Piston, kg	Approximate Pressure	
			kPa	psi
80/2.16	80	2.16		
125/0.325	125	0.325	44.8	6.5
125/2.16	125	2.16	298.2	43.25
150/2.16	150	2.16	298.2	43.25
180/0.325	180	0.325	44.8	6.5
180/2.16	180	2.16	298.2	43.25
180/21.60	180	21.60	2982.2	432.5
200/5.0	200	5.0	689.5	100.0
230/1.2	230	1.2	165.4	24.0
230/3.8	230	3.8	524.0	76.0
265/12.5	265	12.5	1723.7	250.0
275/0.325	275	0.325	44.8	6.5
230/2.16	230	2.16	298.2	43.25
180/1.05	180	1.05	144.7	21.0
180/10.0	180	10.0	1379.0	200.0
300/1.2	300	1.2	165.4	24.0
190/5.0	190	5.0	689.5	100.0
235/1.0	235	1.0	138.2	20.05
235/2.16	235	2.16	298.2	43.25
235/5.0	235	5.0	689.5	100.0
250/2.16	250	2.16	298.2	43.25
310/12.5	310	12.5	1723.7	250.0
210/2.16	210	2.16	298.2	43.25
285/2.16	285	2.16	298.2	43.25
315/5.0	315	5.0	689.5	100.0
372/2.16	372	2.16	298.2	43.25
372/5.0	372	5.0	689.5	100
297/5.0	297	5.0	689.5	100
230/21.6	230	21.6	2982.2	432.5
230/5.0	230	5.0	689.5	100
265/21.6	265	21.6	2982.2	432.5
265/31.6	265	31.6	4361.2	632.5
271.5/2.16	271.5	2.16	298.2	43.25
220/10	220	10.0	1379.0	200.0
250/1.2	250	1.2	165.4	24.0
265/3.8	265	3.8	524.0	76.0
265/5	265	5.0	689.5	100.0

derived by dividing the flow rate at Condition 190/10 by the flow rate at Condition 190/2.16.

NOTE 12—When determining such a ratio of flow rates for a material at the same temperature under different loads, it has been found that precision is maximized when one operator uses one Procedure (A or B), the same plastometer, and the same die for both measurements (the die need not be removed from the plastometer between the two determinations).

#### 9. Procedure A—Manual Operation

9.1 Select conditions of temperature and load from Table 1 in accordance with material specifications such that flow rates will fall between 0.15 to 50 g/10 min.

9.2 Ensure that the bore of the extrusion plastometer is properly aligned in the vertical direction. (See Appendix X1.)

9.3 Inspect the apparatus and die for cleanliness. If it is not clean, see 9.11.

NOTE 13—The degree of cleanliness can significantly influence the flow rate results, therefore a thorough method of cleaning must be established. It has been found that three swabs of the barrel is satisfactory for most materials and that the die, barrel, and piston are more easily cleaned while hot.

9.4 Check the die bore diameter with appropriately sized no-go/go gages prior to testing. Make frequent checks to determine whether the die diameter (tested with die at  $23 \pm 5^\circ\text{C}$ ) is within the tolerances given in 5.3.

NOTE 14—Cleaning and usage can result in a die diameter that is out of specifications. Data has shown that erroneous results will be obtained if the die diameter is not within the appropriate tolerances.

9.5 Verify that the temperature is stable and within  $\pm 0.2^\circ\text{C}$  of the appropriate test temperature as specified in 5.5.1.

9.6 Insert the die and the piston. The temperature of the cylinder with the piston and die in place must be stable at the appropriate test temperature 15 min before testing is begun. When equipment is used repetitiously, it should not be necessary to heat the piston and die for 15 min.

NOTE 15—The reduction in heating time when the unit is being used repetitiously is only allowed when runs of the same or similar material are being measured over a continuous time frame. If the piston and die are removed and cleaned, they should be considered "cold" and the full 15 minutes heating stabilization time required.

9.7 Remove the piston and place it on an insulated surface. Charge the cylinder within 1 min with a weighed portion of the sample in accordance with the expected flow rate, as given in Table 2. Reinsert the piston and add the appropriate weight.

NOTE 16—Experience has shown that for the best reproducibility the piston should operate within the same part of the cylinder for each measurement. The piston is scribed so the starting point for each extrusion is roughly the same. Some excess of material over the minimum required for the actual flow measurement portion of the test is provided by the

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TABLE 2 Standard Test Conditions, Sample Mass,<sup>a</sup> and Testing Time<sup>b</sup>

Flow Range, g/10 min	Suggested Mass of Sample in Cylinder, g	Time Interval, min	Factor for Obtaining Flow Rate in g/10 min
0.15 to 1.0	2.5 to 3.0	6.00	1.67
>1.0 to 3.5	3.0 to 5.0	3.00	3.33
>3.5 to 10	4.0 to 8.0	1.00	10.00
>10 to 25	4.0 to 8.0	0.50	20.00
>25	4.0 to 8.0	0.25	40.00

<sup>a</sup> This is a suggested mass for materials with melt densities of about 0.7 g/cm<sup>3</sup>. Correspondingly, greater quantities are suggested for materials of greater melt densities. Density of the molten resin (without filler) may be obtained using the procedure described by Terry, B. W., and Yang, K., "A New Method for Determining Melt Density as a Function of Pressure and Temperature," *SPE Journal*, SPEJA, Vol. 20, No. 6, June 1964, p. 540 or the procedure described by Zoller, Paul, "The Pressure-Volume-Temperature Properties of Polyolefins," *Journal of Applied Polymer Science*, Vol. 23, 1979, p. 1051. It may also be obtained from the weight of an extruded known volume of resin at the desired temperature. For example, 25.4 mm (1 in.) of piston movement extrudes 1.804 cm<sup>3</sup> of resin. An estimate of the density of the material can be calculated from the following equation:

$$\text{resin density at test temperature} = M/1.804$$

where:

$M$  = mass of extruded resin.

<sup>b</sup> See 9.14.

charging weights shown in Table 2. This is necessary to achieve a void-free extrudate and flow equilibrium before start of rate measurements.

Note 17—It is frequently helpful to take interim cuts of the extrudate at uniform time intervals during the specified extrusion time. Weights of these individual cuts give an indication of the presence of bubbles which may be masked due to their size or to opacity of the sample. This technique is particularly helpful in the case of highly pigmented materials. Forcing out some of the resin manually during the preheat period often eliminates bubbles in the test extrudate.

9.8 Allow time for the material to soften and begin to melt, and then purge some material to a position such that subsequent travel of the piston will position the lower scribe mark at the reference start position  $7.0 \pm 0.5$  min from the completion of the charge. Purge must be completed at least 2 min prior to start of the test for materials having melt flow rates less than 10 g/10 min.

Note 18—It has been found that purging within 60 s of the start time will result in higher variability in the data.

Note 19—There may be cases where the  $7.0 \pm 0.5$  min is too much or not enough preheat time. For those materials, provisions must be in the material documents. It is necessary to refer to the appropriate material document before beginning any test.

Note 20—Additional care may be necessary to prevent thermal degradation in the extrusion plastometer. This is sometimes done by the addition of an appropriate antioxidant. For highly unstable materials, it may be necessary to use alternative techniques as an indication of flow characteristics.

9.9 For materials with flow rates greater than 10 g/10 min, a weight (and if needed, a piston) support must be used after the initial purge. The support shall be removed at such a time as to allow the test to begin within  $7 \pm 0.5$  min of the completion of the charge. The piston/weight support should be of such a length that the lower scribe mark of the supported piston/weight will be 25 mm above the top of the guide bushing or other suitable reference mark.

Note 21—It has been found that the effect of choosing plugging

weight support, or both, is significant to the flow rate results. The choice of piston support was made to cover all conditions and flow rates 10 to 50 g/10 min.

9.10 For all tests, start collecting a timed extrudate when requirements for the piston position are met, provided this is within  $7.0 \pm 0.5$  min from the end of charging; otherwise, discard the charge and repeat the test with readjusted piston position after the initial purge, or change weights. Requirements are that the top scribed mark on the piston be visible above the cylinder or index and that the lower scribe mark be in the cylinder or below the index. As the lower scribed mark approaches the top of the cylinder or index, reset the timer to zero, then simultaneously start the timer and make the initial cut-off when the position requirements are met. Make the final cut-off exactly when the time interval given in Table 2 is reached. Collect the timed extrudate. If the extrudate contains visible bubbles, discard the complete charge and begin the test again.

Note 22—The charge weight should only be increased if no material is being purged and there is still not enough material to complete the test.

9.11 Discharge the remainder of the specimen and push the die out through the top of the cylinder. Swab out the cylinder with cloth patches after the manner of cleaning a pistol barrel. The die may be cleaned by dissolving the residue in a solvent. A better method is pyrolytic decomposition of the residue in a nitrogen atmosphere. Place the die in a tubular combustion furnace or other device for heating to  $550 \pm 10^\circ\text{C}$  and clean with a small nitrogen purge through the die. This method is preferable to flame or solvent cleaning, being faster than solvent cleaning and less detrimental to the die than an open flame. In certain cases where materials of a given class having similar flow characteristics are being tested consecutively, interim die cleaning may be unnecessary. In such cases, however, the effect of cleaning upon flow rate determination must be shown to be negligible if this step is avoided.

9.12 Once the extrudate is cool, weigh to the nearest 1 mg.

9.13 Multiply the weight of the extrudate by the appropriate factor shown in Table 2 to obtain the flow rate in grams per 10 min.

Note 23—Frequently, errors in test technique, apparatus geometry, or test conditions, which defy all but the most careful scrutiny exist, causing discrepancy in flow rate determinations. The existence of such errors is readily determined by periodically measuring a reference sample of known flow rate. The flow rate value and range to be tolerated can be determined using a statistically correct test program composed of multiple determinations with various instruments. Standard samples of polyethylene, linear or branched, are available from the National Institute of Standards and Technology.

9.14 In case a specimen has a flow rate at the borderline of the ranges in Table 2 and slightly different values are obtained at different time intervals, the referee value shall be obtained at the longer time interval.

## 10. Procedure B—Automatically Timed Flow Rate Measurement

### 10.1 Apparatus:

10.1.1 Extrusion plastometer and auxiliary equipment are detailed in Section 4 and below.

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10.1.2 A timing device shall electrically, optically, or mechanically time piston movement within the specified travel range. The requirements of the system are as follows:

10.1.2.1 Sense and indicate the piston travel time within  $\pm 0.01$  s (see Note 1).

10.1.2.2 Measure piston travel within  $\pm 0.4$  % of the nominal preset value (see 10.1.2.4 and 10.1.2.5) for use in the flow rate calculations.

10.1.2.3 Any effects on the applied load must be included in the allowable tolerance given in 5.4.3.

10.1.2.4 It should be preset or be settable for measuring piston travel of  $6.35 \pm 0.25$  mm for flow rates up to 10 g/10 min.

10.1.2.5 It should be preset or be adjustable for measuring piston travel of  $25.4 \pm 0.25$  mm for flow rates greater than 10 g/10 min.

10.1.2.6 To ensure high interlaboratory reproducibility, it is important that the timing device operates within a fixed portion of the cylinder. This is defined as the portion of the cylinder between  $46 \pm 2$  mm and  $20.6 \pm 2$  mm above the top of the die.

10.1.2.7 Check die, cylinder, and position dimensions for conformance to 5.2-5.4 and Figs. 1 and 2.

### 10.2 Procedure:

10.2.1 Refer to Table 1 for selection of conditions of temperature and load in accordance with the material specification.

10.2.2 Check the die bore diameter with appropriately sized no-go/go gages prior to testing. Make frequent checks to determine whether the die diameter (tested with die at  $23 \pm 5^\circ\text{C}$ ) is within the tolerances given in 5.3 (see Note 14).

10.2.3 Ensure that the bore of the extrusion plastometer is properly aligned in the vertical direction (see Appendix X1).

10.2.4 Inspect the apparatus and die for cleanliness. If it is not clean, see 9.11 and Note 13.

10.2.5 Check the die bore diameter with appropriately sized no-go/go gages before beginning the test. Make frequent checks to determine whether the die diameter is within the tolerances given in 5.3 (see Note 14).

10.2.6 Verify that the temperature is stable and within  $\pm 0.2^\circ\text{C}$  of the appropriate test temperature as specified in 5.5.1.

10.2.7 Insert the die and the piston. The temperature of the cylinder with the piston and die in place must be stable at the appropriate test temperature 15 min before testing is begun. When equipment is used repetitiously, it should not be necessary to heat the piston and die for 15 min.

TABLE 3 Factors for Calculation of Flow Rate

Material (Unpigmented)	Tempera- ture, $^\circ\text{C}$	Piston Travel, L, cm [in.]	Factor for Calculation of Flow Rate, $F^a$
Polyethylene	180	2.54 [1]	826
Polyethylene	180	0.635 [0.25]	207
Polypropylene	230	2.54 [1]	799
Polypropylene	230	0.635 [0.25]	200

<sup>a</sup> Factors calculated using melt-density values of  $0.7836 \text{ g/cm}^3$  for polyethylene and  $0.7386 \text{ g/cm}^3$  for polypropylene, as expressed in article by Zoller, Paul, "The Pressure-Volume-Temperature Properties of Polyolefins," *Journal of Applied Polymer Science*, Vol 22, 1979, P. 1051. The base densities at  $23^\circ\text{C}$  for which the melt densities are reported were  $0.917 \text{ g/cm}^3$  for annealed low-density polyethylene and polypropylene homopolymer.

10.2.8 Adjust the travel arm to  $6.35 \pm 0.25$  mm for measuring materials with expected flow rates of up to 10 g/10 min or  $25.40 \pm 0.25$  mm for measuring materials with expected flow rates of 10 g/10 min or higher.

NOTE 24—It has been found that for some materials the melt flow rates obtained on a material will be different depending on which timer length is chosen; therefore, it is important to adhere to the protocol in 10.2.8 to compare interlaboratory results.

10.2.9 Remove the piston and place it on an insulated surface. Charge the cylinder within 60 s with a weighted portion of the sample in accordance with the expected flow rate, as given in Table 2. Reinsert the piston and add weight.

10.2.10 Allow time for the material to soften and begin to melt, and then purge some material to a position such that subsequent travel of the piston will position the lower scribe mark at the reference start position  $7.0 \pm 0.5$  min from the completion of the charge. Purge must be completed at least 2 min prior to start of the test for materials having melt flow rates less than 10 g/10 min (see Note 18).

10.2.11 Weight and piston support, if needed, must be used after the initial purge. The support will be removed at such a time as to allow the timer to activate within  $7.0 \pm 0.5$  min after completion of the charge. If the timer is not activated within  $7 \pm 0.5$  min after the completion of the charge, the test must be repeated with readjusted piston position after the initial purge, or change weights. The piston/weight support should be of such a length that the lower scribe mark of the supported piston/weight will be at least 25 mm above the top of the cylinder. Only use piston support if there is excessive material flow (see Notes 21 and 22).

10.2.12 For materials greater than 50 g/10 min a die plug must be used in addition to the piston/weight support. The die plug is inserted before charge and is removed prior to removing the piston/weight support. The initial charge should be adjusted to reduce excess flow. If the timer arm is not activated within  $7 \pm 0.5$  min after the completion of the charge the test must be repeated with readjusted piston position, or change weights (see Notes 21 and 22). (Warning—Rapid expulsion of material when die plug is removed may be hazardous.)

10.2.13 If the timed extrudate contains visible bubbles, repeat the test (see Note 23).

10.2.14 Record the time to the nearest 0.01 s for the piston to complete the calibrated distance of travel.

10.2.15 Discharge any remaining resin and clean the die, piston, and cylinder as detailed in 9.11.

## 11. Procedure C—Automatically Timed Flow Rate Measurement for High Flow Rate Polyolefins Using Half-Height, Half Diameter Die

### 11.1 Apparatus:

11.1.1 Extrusion plastometer and auxiliary equipment are detailed in Sections 5 and 10.

11.1.2 For polyolefins with a MFR of 75 or greater using the standard die (See 5.3), an alternate die can be used to reduce the flow rate of these materials and improve the reproducibility of results. The alternate die dimensions shall be: Height 4.000

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$\pm 0.025$  mm; bore diameter  $1.048 \pm 0.005$  mm. No spacer shall be used with this die. Bore and finish requirements are the same as 5.3.

11.1.3 For calibration of the temperature indicating device, 5.5.2 shall be used with the variation that temperatures are measured at  $14 \pm 1$  mm and at a nominal 79 mm above the upper surface of the die.

11.1.4 If a thermometer as described in 5.7 is used to indicate temperature, it can be used to monitor indirectly the temperature of the material 14 mm above the upper surface of the die and may be calibrated via 11.1.3.

#### 11.2 Procedure:

11.2.1 Use procedure described in 10.2 with the exception that the die diameter and tolerances are given in 11.1.2.

### 12. Calculation (Procedures B and C)

12.1 Calculate the flow rate in grams per 10 min or volume rate in  $\text{cm}^3$  per 10 min as follows (see Note 23):

$$\text{Flow rate} = (426 \times L \times d)/t$$

or

$$\text{Volume rate} = 426 \times L/t$$

where:

$L$  = length of calibrated piston travel, cm,

$d$  = density of resin at test temperature,  $\text{g}/\text{cm}^3$  (see reference under Table 2),

$t$  = time of piston travel for length  $L$ , s, and

426 = mean of areas of piston and cylinder  $\times 600$ .

Note 25—Factors that may be substituted in the following equation are given for some materials in Table 3.

$$\text{Flow rate, g/10 min} = Ft$$

where:

$F$  = factor from Table 3, and

$t$  = time of piston travel for length  $L$ , s.

12.2 Agreement between Procedures A and B may be optimized if an average melt density for a particular type of material is determined with the actual equipment used and that value is substituted into the equation given in 12.1.

### 13. Procedure D—Multi-Weight Using Automatically Timed Flow Rate Measurement

#### 13.1 Apparatus:

13.1.1 Extrusion plastometer and auxiliary equipment are detailed in Section 4 and below.

13.1.2 A timing device shall electrically, optically, or mechanically time piston movement within the specified travel range. The requirements of the system are as follows:

13.1.2.1 Sense and indicate the piston travel time within  $\pm 0.01$  s (see Note 1).

13.1.2.2 Measure piston travel within  $\pm 0.4$  % of the nominal preset value for use in the flow rate calculations.

13.1.2.3 Any effects on the applied load must be included in the allowable tolerance given in 5.4.3.

13.1.2.4 To ensure high interlaboratory reproducibility, it is important that the timing device operates within a fixed portion of the cylinder. This is defined as the portion of the cylinder between  $46 \pm 2$  mm and  $20.6 \pm 2$  mm above the top of the die.

13.1.2.5 Check die, cylinder, and position dimensions for conformance to 5.2-5.4 and Figs. 1 and 2.

#### 13.2 Procedure:

13.2.1 Refer to Table 1 for selection of conditions of temperature and loads in accordance with the material specification.

13.2.2 Check the die bore diameter with appropriately sized no-go/go gages prior to testing. Make frequent checks to determine whether the die diameter (tested with die at  $23 \pm 5^\circ\text{C}$ ) is within the tolerances given in 5.3 (see Note 14).

13.2.3 Ensure that the bore of the extrusion plastometer is properly aligned in the vertical direction (see Appendix X1).

13.2.4 Inspect the apparatus and die for cleanliness. If it is not clean, see 9.11 and Note 13.

13.2.5 Check the die bore diameter with appropriately sized no-go/go gages before beginning the test. Make frequent checks to determine whether the die diameter is within the tolerances given in 5.3 (see Note 14).

13.2.6 Verify that the temperature is stable and within  $\pm 0.2^\circ\text{C}$  of the appropriate test temperature as specified in 5.5.1.

TABLE 4 Precision, Procedure A (Values in g/10 min)

Material	Condition	Average	$S_p^A$	$S_R^B$	$I_p^C$	$I_R^D$	Number of Laboratories
Polyethylene	190/2.16	0.27	0.008	0.022	0.023	0.063	9
Polyethylene	190/2.16	0.40	0.012	0.038	0.035	0.108	9
Polyethylene	190/2.16	2.04	0.026	0.079	0.073	0.224	9
Polyethylene	180/2.16	44.1	0.919	1.232	2.560	3.488	7
Polypropylene	230/2.16	2.23	0.106	0.226	0.299	0.639	9
Polypropylene	230/2.16	7.09	0.222	0.471	0.627	1.391	9
Polypropylene	230/2.16	32.8	0.581	1.051	1.644	2.974	9
Polystyrene	200/5	1.67	0.024	0.122	0.088	0.344	6
Polystyrene	200/5	8.82	0.190	0.667	0.538	1.886	6
Polystyrene	200/5	13.3	0.305	0.925	0.884	2.617	6
Polycarbonate	300/1.2	2.41	0.076	0.115	0.215	0.326	4
Polycarbonate	300/1.2	10.5	0.429	0.647	1.215	1.890	4
Polycarbonate	300/1.2	16.2	0.155	1.109	0.438	3.140	4
Acrylic	230/3.8	2.59	0.051	0.051	0.145	0.145	3

<sup>A</sup>  $S_p$  = within-laboratory standard deviation of the average.

<sup>B</sup>  $S_R$  = between-laboratories standard deviation of the average.

<sup>C</sup>  $I_p$  =  $2.83 S_p$  and

<sup>D</sup>  $I_R$  =  $2.63 S_R$



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TABLE 5 Precision, Procedure B (Values in g/10 min)

Material	Condition	Average	$S_r^A$	$S_R^B$	$I_r^C$	$I_R^D$	Number of Laboratories
Polyethylene	190/2.16	0.27	0.009	0.014	0.026	0.039	8
Polyethylene	190/2.16	0.40	0.016	0.027	0.045	0.076	8
Polyethylene	190/2.16	2.04	0.040	0.094	0.112	0.266	9
Polyethylene	190/2.16	43.7	0.997	1.824	2.619	5.443	8
Polypropylene	230/2.16	2.25	0.052	0.214	0.1466	0.604	8
Polypropylene	230/2.16	7.16	0.143	0.589	0.4051	1.666	8
Polypropylene	230/2.16	32.6	0.693	0.946	1.959	2.672	8
Polystyrene	200/5	1.65	0.037	0.166	0.106	0.470	4
Polystyrene	200/5	8.39	0.144	0.423	0.406	1.197	4
Polystyrene	200/5	13.0	0.108	0.387	0.306	1.097	4

<sup>A</sup>  $S_r$  = within-laboratory standard deviation of the average.<sup>B</sup>  $S_R$  = between-laboratories standard deviation of the average.<sup>C</sup>  $I_r$  = 2.83  $S_r$  and<sup>D</sup>  $I_R$  = 2.83  $S_R$ .

TABLE 6 Precision, Procedure A (Values in g/10 min)

Material	Condition	Average	$S_r^A$	$S_R^B$	$I_r^C$	$I_R^D$	Number of Labs
PMMA	230/3.8	1.51	0.013	0.066	0.037	0.242	7
LDPE	180/2.16	1.74	0.027	0.052	0.075	0.144	11
Polystyrene	200/5.0	1.88	0.040	0.085	0.112	0.238	9
HDPE	190/2.16	5.36	0.049	0.103	0.137	0.288	11
Polypropylene	230/2.16	10.94	0.088	0.473	0.247	1.324	10
Polycarbonate	300/1.2	13.59	0.109	0.233	0.305	0.653	4 <sup>E</sup>
Acetal	190/2.16	25.30	0.235	0.571	0.658	1.599	7

<sup>A</sup>  $S_r$  = within-laboratory standard deviation of the average.<sup>B</sup>  $S_R$  = between-laboratories standard deviation of the average.<sup>C</sup>  $I_r$  = 2.83  $S_r$ .<sup>D</sup>  $I_R$  = 2.83  $S_R$ .<sup>E</sup> Insufficient laboratories to meet Practice E 691.

TABLE 7 Precision, Procedure B (Values in g/10 min)

Material	Condition	Average	$S_r^A$	$S_R^B$	$I_r^C$	$I_R^D$	Number of Labs
PMMA	230/3.8	1.54	0.026	0.102	0.072	0.288	6
LDPE	190/2.16	1.76	0.015	0.053	0.042	0.149	11
Polystyrene	200/5.0	1.89	0.042	0.102	0.117	0.285	7
HDPE	190/2.16	5.41	0.041	0.113	0.114	0.316	10
Polypropylene	230/2.16	10.56	0.357	0.491	0.989	1.376	10
Polycarbonate	300/1.2	13.79	0.104	0.477	0.282	1.335	3 <sup>E</sup>
Acetal	190/2.16	25.64	0.182	0.822	0.508	2.302	6

<sup>A</sup>  $S_r$  = within-laboratory standard deviation of the average.<sup>B</sup>  $S_R$  = between-laboratories standard deviation of the average.<sup>C</sup>  $I_r$  = 2.83  $S_r$ .<sup>D</sup>  $I_R$  = 2.83  $S_R$ .<sup>E</sup> Insufficient laboratories to meet Practice E 691.

TABLE 8 Precision, Procedure B (Values in g/10 min)

Material	Condition	Average	$S_r^A$	$S_R^B$	$I_r^C$	$I_R^D$
Polypropylene	230/2.16	245	13.2	16.6	37.4	46.9
Polypropylene	230/2.16	462	31.8	40.0	89.9	113
Polypropylene	230/2.16	837	20.9	58.6	59.1	166
Polypropylene	230/2.16	1603	129	243	365	688

<sup>A</sup>  $S_r$  = within-laboratory standard deviation of the average.<sup>B</sup>  $S_R$  = between-laboratories standard deviation of the average.<sup>C</sup>  $I_r$  = 2.83  $S_r$  and<sup>D</sup>  $I_R$  = 2.83  $S_R$ .

13.2.7 Insert the die and the piston. The temperature of the cylinder with the piston and die in place must be stable at the appropriate test temperature 15 min before testing is begun. When equipment is used repetitiously, it should not be necessary to heat the piston and die for 15 min.

13.2.8 Remove the piston and place it on an insulated surface. Charge the cylinder within 60 s with a weighted

portion of the sample as given in Table 2 for the expected flow rate at the first test load.

13.2.9 Allow time for the material to soften and begin to melt, and then purge some material to a position such that subsequent travel of the piston will position the lower scribe mark at the reference start position  $7.0 \pm 0.5$  min from the completion of the charge. Purge must be completed at least 2 min prior to start of the test for materials having melt flow rates less than 10 g/10 min (see Note 18).

13.2.10 Weight and piston support, if needed, must be used after the initial purge. The support will be removed at such a time as to allow the timer to activate within  $7.0 \pm 0.5$  min after completion of the charge. If the timer is not activated within  $7 \pm 0.5$  min after the completion of the charge, the test must be repeated with readjusted piston position after the initial purge, or change weights. The piston/weight support should be of

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such a length that the lower scribe mark of the supported piston/weight will be at least 25 mm above the top of the cylinder. Only use piston support if there is excessive material flow (see Notes 21 and 22).

13.2.11 If the material flows from the die when both the piston and weight are supported, a die plug must be used. The die plug is inserted before charge and is removed prior to removing the piston/weight support. The initial charge should be adjusted to reduce excess flow. If the timer arm is not activated within  $7 \pm 0.5$  min after the completion of the charge the test must be repeated with readjusted piston position, or change weights (see Notes 21 and 22). (Warning—Rapid expulsion of material when die plug is removed may be hazardous.)

13.2.12 If the timed extrudate contains visible bubbles, repeat the test (see Note 23).

13.2.13 Record the times to the nearest 0.01 s for a minimum of two piston travel distances for each test load. Piston travel distances shall be chosen so that these times are at least 2 s and that the distances can be determined within  $\pm 0.4\%$ .

13.2.14 After at least two measurements have been taken at the initial load condition, change to the next test load and allow for sufficient piston travel or time to achieve stable flow before recording the piston travel times at this load. Do not take readings below 18.6 mm above the top of the die.

NOTE 26—Normally 5 mm of piston travel or 2 minutes is sufficient to obtain stable flow after changing test loads.

13.2.15 Repeat the above for any additional test loads. Do not take any readings below 18.6 mm above the top of the die.

13.2.16 Discharge any remaining resin and clean the die, piston, and cylinder as detailed in 9.11.

### 13.3 Calculations:

13.3.1 Calculate the individual flow rate values in accordance with Section 12 (Procedure B).

13.3.2 If the differences between the individual flow rates at each test load is no greater than 3 %, calculate the average flow rate for each test load. If differences are  $>3\%$  repeat the test. To achieve stable flow after applying each test load, it may be necessary to increase the time or the piston travel distance before taking a measurement.

13.3.3 Calculate the Flow Rate Ratio (FRR) as the ratio of the average flow rate at the higher load to the average flow rate at the lower load.

## 14. Report

14.1 Report the following information:

14.1.1 Statement indicating the nature and physical form of the material charged to the cylinder.

14.1.2 Temperature and load at which the test is run shall be reported. The results and test conditions can be referred to as FR-condition, where the standard designation for the condition from Table 1 is shown (for example, FR-190/2.16).

NOTE 27—It has become customary to refer to the flow rate of polyethylene as "melt index" when obtained under Condition 190/2.16. However, for all other materials the use of melt index or any term other than "flow rate" is discouraged, regardless of the condition used.

14.1.3 Flow rate reported as the rate of extrusion in grams per 10 min or volume rate in  $\text{cm}^3$  per 10 min.

14.1.4 Procedure used (A, B, or C).

14.1.5 Any unusual behavior of the test specimen such as discoloration, sticking, extrudate surface irregularity or roughness, etc.

14.1.6 Details of conditioning, if any.

14.1.7 For multi-weight tests using Procedure D, also report:

14.1.7.1 The average flow rate at each test load.

14.1.7.2 The Flow Rate Ratio (FRR) together with the temperature and loads used (for example, FRR-190/2.16/2.16).

14.1.7.3 Whether the decreasing or increasing load technique was used.

## 15. Precision and Bias (Procedures A, B, and C)

### 15.1 Precision:

15.1.1 Tables 4 and 5 are based on a round robin<sup>6</sup> conducted in 1986 and 1987, involving polypropylene, polyethylene, polystyrene, polycarbonate and acrylic materials. Tables 6 and 7 are based on a round robin<sup>7</sup> completed in 1997 involving low and high density polyethylene, polypropylene, polystyrene, polycarbonate, PMMA, and acetal. The number of participating laboratories is shown for each material. Data for Tables 4 and 5 were generated through each lab testing two specimens for each material on three different days, while data for Tables 6 and 7 were generated through each lab testing two specimens for each material on two different days. The analysis in Practice E 691 is based on a test result being the average of two specimens.

15.1.2 Table 8 is based on a round robin<sup>8</sup> conducted in 1980 using Procedure B. Four polypropylene samples having flow rates from 250 to 1500 were tested in nine laboratories. (Warning—The following explanations of  $I_L$  and  $I_R$  (15.1.4-15.1.6) are only intended to present a meaningful way of considering the approximate precision of this test method. The data in Tables 4-8 should not be vigorously applied to acceptance or rejection of material since those data are specific to the round robin and may not be representative of other lots, conditions, materials or laboratories. Users of this test method should apply the principles outlined in Practice E 691 to generate data specific to their laboratory and materials. The principles of 15.1.4-15.1.7 would then be valid for such data.)

15.1.3 Table 9 is based on a round robin conducted in 1999 on Procedure C. Data for seven of the eight participating laboratories were included in the statistics for this table. Four polyethylene materials were tested with melt flow rates using the standard die ranging from approximately 35 to 2350 g/10 min using the half-height, half-diameter die.

15.1.4 Concept of  $I_L$  and  $I_R$ —Relevant if  $S_L$  and  $S_R$  have been calculated from a large enough body of data, and if test results are averages obtained from testing two specimens.

<sup>6</sup> Supporting data are available from ASTM Headquarters. Request RR: D20 1164.

<sup>7</sup> Supporting data are available from ASTM Headquarters.

<sup>8</sup> Supporting data are available from ASTM Headquarters. Request RR: D20 1124.

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TABLE 9 Precision Data for High Melt Flow Polyolefins  
Procedure C (Values in g/10 min)<sup>a</sup>

Materials <sup>b</sup>	Average	$S_r$	$S_R$	$r$	$R$
PE-A (35)	4.67	0.068	0.119	0.181	0.334
PE-B (185)	24.30	0.688	1.168	1.928	3.270
PE-C (2350)	315.7	10.81	19.89	30.27	55.69
PE-D (122)	16.20	0.188	0.348	0.526	0.975

<sup>a</sup>This data was generated with dies having a nominal length of 3.985 mm versus the required 4.000 mm.

<sup>b</sup>Numbers in parentheses are approximate melt flow rate values of materials using standard die (5.3).

15.1.5 *Repeatability,  $I_r$* —In comparing two test results for the same material, obtained by the same operator using the same equipment on the same day, the two test results should be judged not equivalent if they differ by more than the  $I_r$  value for that material.

15.1.6 *Reproducibility,  $I_R$* —In comparing two test results for the same material, obtained by different operators using different equipment on different days, the two test results should be judged not equivalent if they differ by more than the  $I_R$  value for that material.

15.1.7 Any judgment in accordance with 15.1.4 and 15.1.6 would have an approximate 95 % (0.95) probability of being correct.

15.2 *Bias*—There are no recognized standards by which to estimate bias of this test method.

## 16. Precision and Bias (Procedure D)

## 16.1 Precision

16.1.1 Procedure D used as many as nine laboratories, five materials and two determinations in order to come up with the following tables of precision supporting the method. This meets or exceeds all the standards required for Practice E 691.

16.1.2 The degree of precision is quite high for all levels of loads on the ram. The highest level of precision was attained for the 2.16 kg load, the lowest for the 21.6 kg load. It is important to note that the 21.6 kg load is not optimum for the short [0.316 in.] die because the speed of testing does not allow

for the flow to reach probably equilibrium rate. All such testing at 21.6 kg is much better done with a longer die such as used in Test Method D 3364. Refer to Table 10 for 2.16 kg load; Table 11 for 5 kg load; Table 12 for 10 kg load and Table 13 for 21.6 kg load.

TABLE 10 Precision Statement for Test Method: 2.16 kg

Materials	Average	$S_r$	$S_R$	$r$	$R$
PE1	0.7567	0.0079	0.0323	0.0220	0.0904
PE2	0.4288	0.0060	0.0277	0.0168	0.0776
PE3	0.5527	0.0096	0.0179	0.0268	0.0502
PE4	0.2827	0.0043	0.0182	0.0120	0.0511
PE5	0.4104	0.0079	0.0119	0.0222	0.0334

TABLE 11 Precision Statement for Test Method: 5 kg

Materials	Average	$S_r$	$S_R$	$r$	$R$
PE1	3.0561	0.7715	0.7809	2.1602	2.1865
PE2	1.9980	0.0483	0.0927	0.1353	0.2595
PE3	1.5821	0.0280	0.0482	0.0729	0.1293
PE4	0.9976	0.2158	0.2560	0.6042	0.7168
PE5	1.2308	0.0170	0.0285	0.0475	0.0798

TABLE 12 Precision Statement for Test Method: 10 kg

Materials	Average	$S_r$	$S_R$	$r$	$R$
PE1	11.6841	0.3065	0.4595	0.8581	1.2867
PE2	7.5768	0.1266	0.2751	0.3544	0.7704
PE3	4.1181	0.0535	0.1036	0.1497	0.2902
PE4	3.7693	0.0538	0.1125	0.1507	0.3151
PE5	3.3471	0.0228	0.0647	0.0638	0.1812

TABLE 13 Precision Statement for Test Method: 21.6 kg

Materials	Average	$S_r$	$S_R$	$r$	$R$
PE1	59.9335	2.1012	2.7667	5.8833	7.7469
PE2	36.2357	1.4450	1.4450	4.0459	4.0459
PE3	14.1078	0.1662	0.3362	0.4653	0.9387
PE4	17.6132	0.3324	0.4258	0.9308	1.1924
PE5	12.2652	0.1104	0.4590	0.3090	1.2851

## 17. Keywords

17.1 melt flow rate; melt index; volume flow rate

## APPENDIXES

(Nonmandatory Information)

## X1. EXTRUSION PLASTOMETER BORE ALIGNMENT

X1.1 A fixture consisting of a circular level mounted on a shaft having two bearing points  $9.47 \pm 0.00 - 0.0076$  mm in diameter that can be inserted into the bore has been found suitable. A circular level that can be rigidly mounted on the

piston rod for insertion into the bore may also be satisfactory. A circular level having a sensitivity of 20 min/2.5 mm has been found satisfactory. Other alignment techniques that give comparable alignment sensitivity would be considered satisfactory.